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Lubrication

A Technical Publication Devoted to
the Selection and Use of Lubricants

THIS ISSUE

LUBRICATING OIL
ANALYSIS



PUBLISHED BY
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LUBRICATION

A TECHNICAL PUBLICATION DEVOTED TO THE SELECTION AND USE OF LUBRICANTS

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LUBRICATING OIL ANALYSIS

FROM the very beginning of the petroleum industry, analysis and testing have been a very important adjunct to the manufacture and use of petroleum lubricants. Numerous pertinent tests enable the supplier to keep constant check on quality through each step of the manufacturing process. Such surveillance also insures a finished product that is dependable and uniform, and inspires confidence in the purchaser that the product will meet the needs of his machine with respect to type of lubricant and duration of service.

A volume of many pages would be required to describe all the tests used on lubricants. This article is concerned primarily with the more common analytical methods of interest to those who use lubricating oils. Correlation between tests will be discussed along with the significance of test results.

Some test methods used widely for lubricating oils are outlined on pages 6 and 7. Since it would be impractical to include all the factors affecting the hundreds of different oils in use today, only the more common factors are given under the column captioned — "Factors Which Usually Affect Test Results". Those listed under "Unused Lubricants" generally apply also to "Used Lubricants" although the reverse is not necessarily true.

In order that the reader retain the correct perspective, the part played by laboratory testing in the development of a new product should be stated. Even though many test methods will help to predict

the behavior of a product in use, the final criterion of suitability of the product must be obtained from multiple tests run on full scale equipment. This equipment should be either the same as that for which the product is intended or should simulate it very closely, with correlation of results previously established.

A description of the common laboratory physical and chemical tests which usually are applied to petroleum lubricants follows. Later on the significance of analytical methods as applied to various phases of petroleum technology is discussed.

TESTS USUALLY ENCOUNTERED

Sensory

In the analysis of a new petroleum product, a sensory examination is always helpful. Appearance and color of the product is a clue to the identity of an oil, or they may indicate contamination. The odor of the sample readily discloses the presence of fatty oils and other additives. The texture or fluidity may suggest the presence of certain thickening agents.

Viscosity

Undoubtedly the most important property of a lubricating oil is viscosity. Therefore it is not surprising to find that viscosity measurement is the test performed most frequently in the laboratory.

The viscosity of a lubricating oil is defined as its resistance to flow. Since this quality varies with temperature, results usually are reported at 100, 130 or 210°F. In the usual test procedures, the time in seconds is measured for a given amount of oil to flow through a finely calibrated orifice.

The thickness of the lubricating film between moving surfaces, as well as the power losses due to internal friction, vary directly with viscosity. For efficient operation it is necessary to maintain a hydrodynamic film and, at the same time, hold down power losses which increase as viscosity rises.

The Saybolt Universal Viscometer is illustrated in Figure 1. The time required for 60 ml. of oil to flow through an orifice of 0.1765 cm., I.D. is measured. Due to the turbulence effect, this procedure is not accurate for oils having flow time less than 32 seconds. For greater accuracy, particularly when it is necessary to estimate viscosities at temperatures other than the test temperature, a capillary-type kinematic viscometer illustrated in Figure 2 has been adopted.

Viscosity Index (V.I.) is an expression of the change in viscosity with change in temperature. The fluidity of a high V.I. oil changes relatively less with temperature change than does the fluidity of a low V.I. oil.

Pour

Another property of lubricating oil related to viscosity is its fluidity at low temperatures. The temperature at which an oil just barely flows, under certain prescribed conditions, is known as its Pour Point. The ASTM Pour Point apparatus is illustrated in Figure 3. Refrigeration oils and products intended for use in cold climates must of necessity have very low Pour Points. However, it is commonly known that an oil in bulk can be poured at temperatures lower than its Pour Point and can be pumped at even lower temperatures.

Gravity

The gravity of an oil is a numerical index of the weight per unit volume. Two scales employed in the petroleum industry are API Gravity and Specific Gravity. The latter is the ratio of the weight of a volume of oil to the weight of an equal volume of water, both at 60°F. and also both corrected for the buoyant effect of air. The API scale is used most often and is related to Specific Gravity as follows:

$$^{\circ}\text{API} = \frac{141.5}{\text{Specific Gravity}} - 131.5$$

In numerical magnitude there is reciprocal relation between API and Specific Gravity, that is, the higher the API the lower the Specific Gravity. Grav-

ity (API) is determined from the depth to which a hydrometer sinks in the sample. Such a hydrometer, with a 0 to 130°F. thermometer, is illustrated in Figure 4.

The API Gravity at the observed temperature can be converted to the API Gravity at standard temperature of 60°F. by reference to prepared charts. Since petroleum products generally are sold basis volume at 60°F., the gravity converted to this temperature is important in transfer calculations.

Gravity, in conjunction with other characteristics, is a clue to the source of the oil, its paraffinic or naphthenic nature and sometimes is an indication of whether it was solvent refined.

Dilution

Dilution of automotive crankcase oil by unburned or partially burned gasoline is determined by adding a large excess of water to the oil and distilling the mixture. The fuel (diluent) which distills over with the steam, is condensed, collected and measured in the specially designed trap illustrated in Figure 5.

Foam

One of the more popular foam tests is ASTM D-892-46T, in which a sample maintained at 75°F. is blown with air for five minutes, then allowed to settle for 10 minutes, the volume of foam being measured at the end of both periods. A second sample at 200°F. is air blown and then after the foam collapses it is air blown at 75°F. Indications are thus obtained of the maximum foam build-up and foam stability at both moderate and elevated temperatures.

Carbon Black Dispersion

In Diesel and gasoline engine service, lubricating oil is contaminated with fine particles of carbon from the combustion process. A crude indication of the ability of a lubricating oil to disperse this carbon is obtained by simple laboratory tests in which finely divided carbon is mixed with the oil alone or with solvent. The mixture is centrifuged or allowed to settle, and the concentration of carbon remaining suspended is measured by opaqueness or weight determination.

Flash and Fire Point

Flash Point is the temperature to which a product must be heated before it evolves sufficient vapor to form a mixture with air which, when brought in contact with a flame, ignites but doesn't continue to burn. If the product is raised to a still higher temperature, the Fire Point is reached at which the inflammable vapor-air mixture, if ignited, continues to burn. The Flash Point of a lubricating oil is determined for purposes of safety in hand-

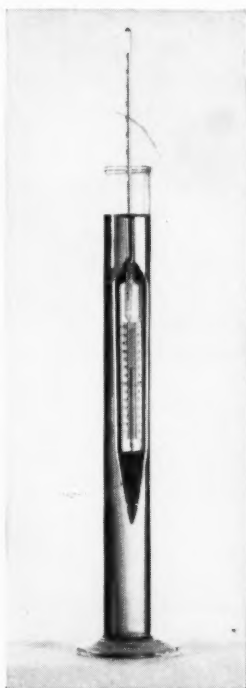


Figure 4 — API Hydrometer with Thermometer.



Figure 3 — ASTM Pour Point Apparatus.



Figure 5 — Specially Designed Trap for Dilution Determination.

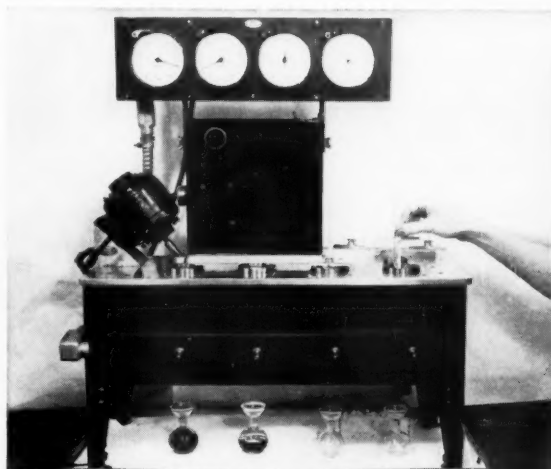


Figure 1 — Saybolt Universal Viscometer.

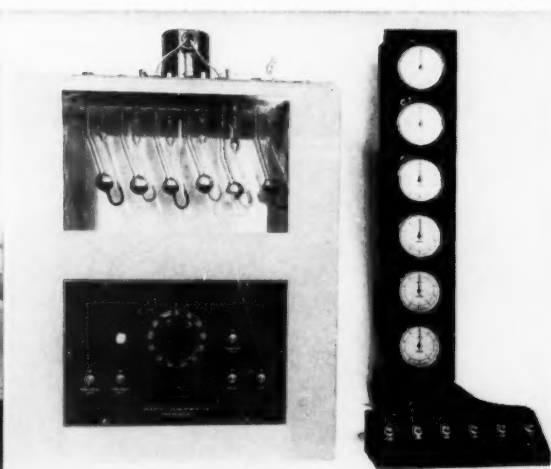


Figure 2 — Capillary Type Kinematic Viscometer.

TABLE I
Comparative Color Scales

Lovibond Color 6" Cell	Color 1/2" Cell	Tag- Robinson Color	ASTM & NPA Color	NPA Color Names
Up to 17.5		21	1	Lily White
17.5 - 60		17 1/2	1 1/2	Cream White
60.5 - 148		12 1/2	2	Extra Pale
149 - 240		10	2 1/2	Extra Lemon Pale
241 - 334		9 3/4	3	Lemon Pale
335 - 474		9	3 1/2	Extra Orange Pale
	44 - 58	8 1/4	4	Orange Pale
	59 - 74	5	4 1/2	Pale
	75 - 100	3 1/2	5	Light Red
	101 - 138	2 1/4	6	Dark Red
	139 - 185	2	7	Claret Red
	186 plus	1	8	—



Figure 6 - Cleveland Open Cup Flash Test Apparatus.

ling and to establish the presence of certain types of contaminants. The Fire Point is used less frequently because it adds little useful information beyond that gained from the Flash Point. The Cleveland Open Cup Flash Tester is illustrated in Figure 6.

Color

The color of petroleum oils is expressed in numerical units which generally are limited to a determination by transmitted light. Color numbers by Saybolt and Tag-Robinson colorimeters are derived from the depth of oil required to match a given color standard; whereas a fixed depth of oil is matched by color standards in the ASTM Union and Lovibond instruments. Table I gives a correlation of values for several color scales in common use.

Neutralization Number

The results of this test are expressed as the milligrams of potassium hydroxide required to neutralize one gram of oil. Its actual significance, which initially was intended as a measure of acidity or alkalinity, has changed to a considerable extent in recent years. At first the test was employed to insure that no mineral acidity from refining operations remained in the oil. The change in significance of the neutralization number has been due principally to two things. First, many of the older acid treating refining procedures have been improved enormously or have given way to non-acidic solvent refining processes. Secondly, since potassium hydroxide used as titrant reacts with many neutral additives which

are compounded in lubricants, the neutralization number of such products is not indicative of corrosive acids.

The two ASTM methods described in the centerspread are similar in that the oil sample is dissolved in benzene and isopropyl alcohol containing 0.5 per cent water. In method ASTM D974-53T, the end point is determined by use of a color indicator; whereas in ASTM D664-52, the end point is determined potentiometrically by means of an electrometric instrument such as that illustrated in Figure 7.

During the past several years many methods¹ with various versions have been suggested. In addition to those outlined in the centerspread, two neutralization procedures used extensively are the ASTM D-188-27T and the "Salt" method. The former was the only ASTM method from 1927 until 1942. It employs phenolphthalein as indicator and alcohol and water in equal volumes as the solvent. The "Salt" method is similar except that naphtha and 25 mls of a saturated sodium chloride solution are added. The neutral salt solution speeds up separation of the oil and aqueous layers, thus enabling the operator to see more quickly the end point which is difficult to observe in dark colored samples.

Figure 8 illustrates neutralization numbers by four methods obtained on four unused additive oils designed for heavy duty service in either gasoline or Diesel engines. Each of these four methods has found prominent use in the petroleum industry. The base oils of these finished samples were highly refined and neutral. The Supplement One oil (A) showed an initial alkaline reaction, hence required the addition of hydrochloric acid to obtain an alkaline neutralization number by three of the methods.

¹H. P. Ferguson. Anal. Chem. 22, 289 (1950)



Figure 7 — Recordomatic Titrometer.

By the ASTM methods the neutralization values tended to be considerably higher than in other procedures. These differences emphasize the importance of proper choice of method and the difficulties of interpretation.

Saponification Number

Like the neutralization number, the units for expressing the saponification number of an oil are the number of milligrams of potassium hydroxide consumed by one gram of oil. Its test conditions differ in that the sample, solvent and excess alkali are refluxed for 30 minutes before titrating. This heating with alkali saponifies fatty materials and is the basis for their determination when they are blended with mineral oils.

Oxidation

Many tests are available for determining the oxidation stability of lubricants, since instability may

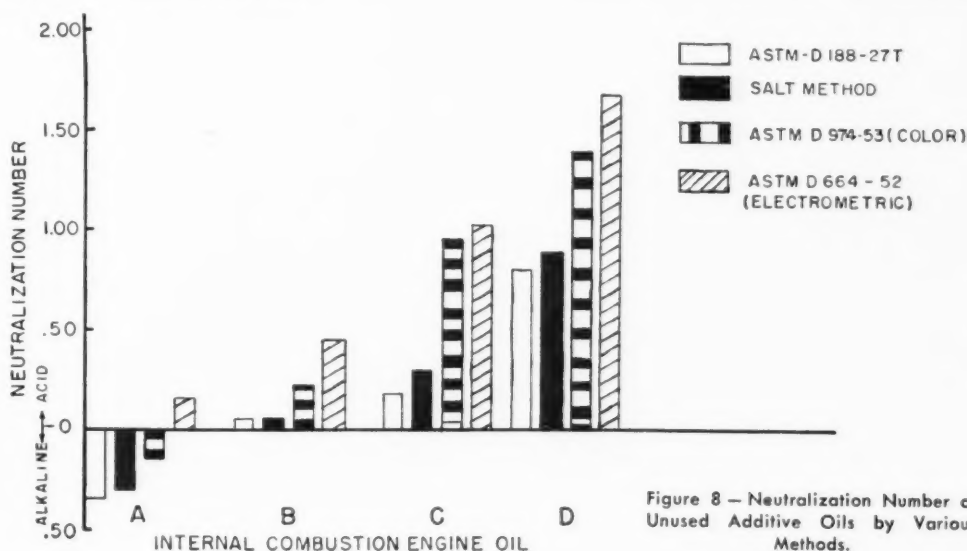


Figure 8 — Neutralization Number of Unused Additive Oils by Various Methods.

LABORATORY METHODS FOR LUBRICATING OILS

Name	ASTM Designation	Method Summary	Factors Which Usually Affect Test Results	
			Unused Lubricants	Used Lubricants
Viscosity		(Test conditions controlled in each method)		
Saybolt Universal (seconds)	D 88-53	Time in seconds for fixed volume of oil to flow thru orifice.	1 Molecular weight and type of hydrocarbons 2 Viscosity index improvers 3 Pour Depressants 4 Thickening agents 5 Detergent and oxidation inhibitors etc.	1 Dilution by fuels 2 Oxidation products 3 Contamination
Kinematic (centistokes)	D 445-53T	Time for fixed volume of oil to flow thru capillary. Results reported in centistokes.		
Pour (°F.)	D 97-47	Lowest temperature at which oil will flow.	1 Pour point additives 2 Paraffin wax 3 Vis. at low temperature	1 Dilution by fuels 2 Oxidation 3 Contamination
Gravity (°API)	D 287-53T	Immersed hydrometer is read, corrected for temperature and reported in degrees API	1 Type of hydrocarbons — paraffinic, naphthenic, etc.	1 Dilution by fuels 2 Contamination
Color				
Tag-Robinson (Color Number)		Depth of oil to match a given color standard	1 Degree and nature of refining	1 Oxidation products 2 Contamination 3 Cleanliness of oil system
Union (Color Number)	D 155-45T	Fixed depth of oil is matched against standard color disc.	1 Degree and nature of refining	
Flash (°F.)	D 92-52	Lowest temperature at which vaporization in air is sufficient to ignite with flame. Water insoluble distillate carried over in a steam distillation.	1 Type and degree of refining	1 Dilution by fuels
Dilution (%)	D 322-35		Not applied to unused samples	1 Gasoline introduced by faulty condition of engine piston-cylinder system
Foam (ml.)	D 892-46T	Oil is air blown and volume of foam after blowing and after settling is reported.	1 Anti-foaming additives 2 Metallo-organic additives	1 Contamination

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Neutralization No. (mgs. per gm.)	D 974-53T (Colorimetric) D 664-52 (Potentiometric)	Sample dissolved in benzene, isopropyl alcohol, and small amount of water is titrated with alcoholic base or acid solution. End point is determined by use of color indicator (D 974) or potentiometrically (D 664)	1 Organic or inorganic acids or bases 2 Metallo-organic additives	1 Presence of additives 2 Acidic oxidation products 3 Contaminants
Ash (%)	D 482-46	Sample is burned, ignited and residue weighed.	1 Metallo-organic additives	1 Lead from fuel 2 Engine wear and corrosion 3 Contamination
Carbon Residue (%)	D 189-52 D 524-52T	Sample is reduced to a carbonaceous residue by evaporating with air excluded and pyrolysis of the vapor.	1 Ash 2 Type of hydrocarbons paraffinic or naphthenic etc.	1 Oxidation 2 Overheating 3 Contaminants
Solubility Precipitation No. (ml. per 10 ml.)	D 91-52	Solubility of the sample in specified solvent is determined.	1 Extraneous Materials	1 Oxidation 2 Overheating 3 Contaminants 4 Water
Pentane and Benzene Insolubles (%)	D 893-52T			
Oxidation (Hours)	D 943-53T	Oxygen is bubbled thru a turbine oil at 95°C. in presence of water and iron-copper catalysts until a specified Neut. No. is reached.	1 Oxidation inhibitors 2 Degree of refining 3 Origin of the oil	1 Oxidation products 2 Contamination 3 Residual inhibitor
Rusting Test	D 665-53T	Steel specimens are immersed in oil at 140°F. and stirred with distilled or sea water. Oil fails the test if rust spots appear on specimen.	1 Rust inhibitors 2 Degree of refining	1 Residual inhibitor 2 Oxidation 3 Contamination

result in formation of sediments, sludges and corrosive products.

A method used extensively is ASTM D943-53T, the "Oxidation Characteristics of Inhibited Steam Turbine Oils", in which the sample, with water and an iron-copper catalyst, is maintained at a temperature of 95°C. while a stream of oxygen flows through the mixture. The course of oxidation is followed by neutralization number tests on samples drawn periodically during the time required to reach a predetermined neutralization number, 2.0 being a common criterion of test termination. In the various popular versions of the Indiana Oxidation Test,² the oil is maintained at elevated temperatures while a stream of air is blown through the sample. The course of oxidation usually is followed by measurement of naphtha insolubles, viscosity or neutralization number.

Rusting

In the ASTM D665-53T procedure for determining the Rust-Preventing Characteristics of Steam Turbine Oil, a steel cylindrical specimen is immersed in a mixture of oil and distilled or sea water which is stirred at a temperature of 140°F. After 24 hours the condition of the metal specimen with respect to rusting is observed.

Corrosion

The MacCoull Corrosion Test³ employs a ratio of oil volume to bearing surface similar to that found in full scale engines and operates under dynamic conditions. The apparatus is shown in Figure 9. Good correlation with multi-cylinder engine bearing behavior has been observed.

Emulsion

The tendency of a lubricating oil to emulsify with water or other aqueous solutions is measured by various emulsion tests. In the popular ASTM test "Steam Emulsion of Lubricating Oils" a sample is blown with steam for five minutes and the time in seconds for the oil to separate is noted. The method⁴ for Demulsibility of Lubricating Oils involves stirring one volume of oil with about two volumes of water under specified conditions of temperature and speed for five minutes. The container is then allowed to stand at test temperature and the maximum rate of oil separation in milliliters per hour is reported.

As the available emulsion tests are not entirely satisfactory since correlation with service is poor, various ASTM committees are engaged in making them more informative.

²T. H. Rogers and B. H. Shoemaker Ind. Eng. Chem. Anal. Ed. 6, 419, (1934).

³MacCoull, N., Ryder, E. A. and Scholp, A. C., SAE Journal (Trans.), 50, 338 (1942).

⁴Demulsibility Test for Lubricating Oils VV-L-791C Method 320.32 (no longer in VVL-book 791E).

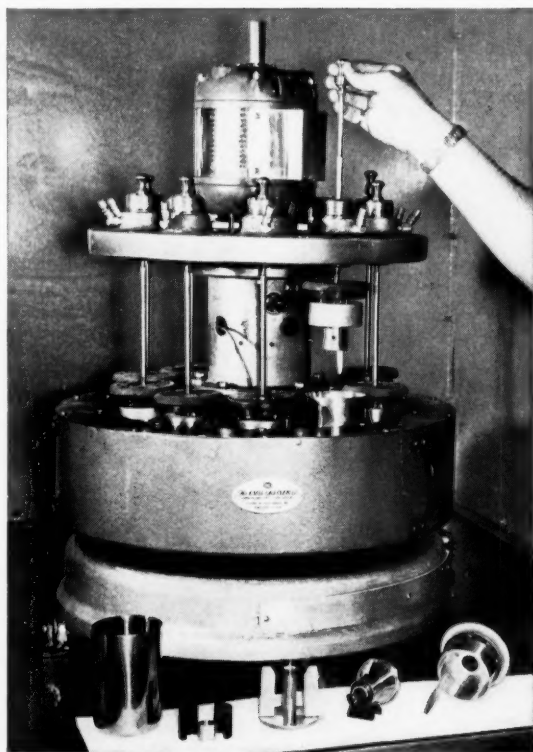


Figure 9 — MacCoull Corrosion Test Apparatus.

Ash

In the ASTM ash test the oil is burned to a carbonaceous residue which is then ignited until it is free of carbon. The ash result is an indication of the amount of metals in an oil. Usual sources of the metals are the metallo-organic additives, tetraethyllead in gasoline or the wearing of various engine parts. Dirt and other contaminants also increase the ash content.

ASTM D874-51, which determines the sulfated residue from a lubricating oil, gives a more accurate estimation of certain metals which are quantitatively converted to their sulfate salt rather than to a mixture of salts such as oxide, sulfate, phosphate or carbonate, as in the ordinary ash procedure. For a variety of metals, the optimum ignition temperature was found to be $775 \pm 25^\circ\text{C}$. However some sulfated residues such as zinc and aluminum are unstable at this temperature and therefore give erratic results.

Carbon Residue

The residue left after evaporation and pyrolysis of an oil in the absence of air is termed "Carbon Residue". It is related crudely to the magnitude of coke-like deposits which may form in engines. However it has lost considerable of its original significance because many modern lubricating oils of out-

standing performance contain additives which leave an ash that is included in the carbon residue result.

SIGNIFICANCE OF THE ANALYSIS

When considering the significance of chemical analysis, physical tests, and other laboratory measurements on lubricants — in fact, on any product — it is important to keep in mind the purpose or objective.

Although chemistry is basically a logical science, it is dangerous to reason exclusively by analogy. For example, if one heats paraffin wax to an elevated temperature and blows air through it for a prolonged time, a liquid is obtained which contains a large concentration of oxygen. With this information as background, one who reasoned exclusively by analogy would expect that if flux oil be heated to a high temperature and blown with air, there would likewise result a liquid product high in oxygen — but this is not the case. Flux oil so treated produces a solid asphalt, which is found to contain little combined oxygen, generally less than a few per cent.

For ease in discussion, the significance of analysis of lubricating oils is considered in relation to three phases of petroleum technology. They are:

1. Research and Development.
2. Manufacture and Control.
3. Product Application.

Research and Development

The scientist engaged in research and development — aware of the hazards of analogy — doesn't presume that a satisfactory product resulted simply because his intended formulation was followed. Since his objective is fundamental information on composition and properties, the finished oil must be checked by a variety of laboratory analyses and tests. A great deal of fundamental information on new products is obtained in the analytical laboratory by elemental analysis to determine sulfur, chlorine, phosphorus, nitrogen, oxygen, carbon, and hydrogen. Other procedures measure functional groups in a product such as carboxyl, carbonyl, and hydroxyl groups.

During the development of new products, numerous physical tests are employed to indicate whether they will be suitable under the conditions for which they are intended. For example, oils intended for either very high or low temperature systems must be controlled carefully in viscosity or pour point. Other bench tests indicating probable performance characteristics are those measuring foam, dispersion, corrosion, oxidation and emulsion.

When a new product is developed, it obviously is most impressive and convincing to evaluate it for performance characteristics in a large number

of units of full scale equipment. Generally, absolute reliance is not placed on the product's behavior in a single full scale device, because modern machines, by their very complexity, sometimes differ in performance from unit to unit. Before a new lubricant is field tested, it must first qualify in reduced and simplified laboratory models such as a single cylinder engine.

In the examination of a newly developed product, one cannot blindly or mechanically apply the tests which may have had significance on previous products. A glaring example occurred several years ago with the introduction of a new product. Neutralization Number, by the then ASTM method, had been in use for many years as a specification for straight mineral oils and, by some, as the principal criterion for deciding when an oil change should be made. Then came oxidation and corrosion resistant detergent oils containing metal additives which, though actually neutral in themselves, caused a fictitiously high neutralization number. Thus unused oils containing zinc additives, for example, would have a neutralization number as high as 2, though no free acid actually was present. This resulted in the exclusion of superior oils which had been proven extensively by actual service performance.

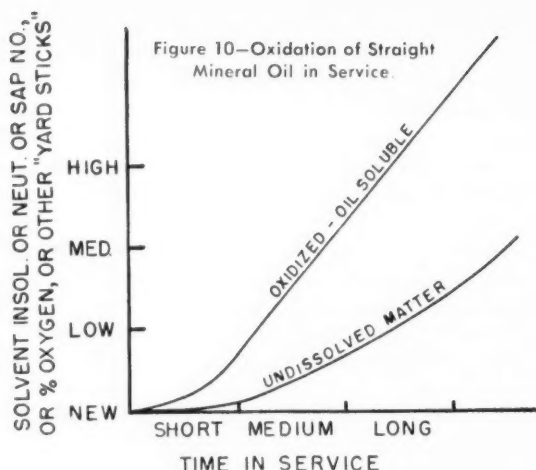
Manufacture and Control

After the satisfactory nature of a product has been established in the laboratory and by field performance in units for which it is intended, subsequent batches may be controlled in manufacturing operations by bench type evaluations, chemical analyses and physical measurements which characterize the product. This, in effect, establishes by simple, economical laboratory tests, whether the batch has the same characteristics — within reasonable limits — as that prior batch which was proven to be satisfactory by the more expensive laboratory and field examination.

Certain relatively simple tests serve to control manufacturing operations of lubricating oils. Among these are gravity, viscosity, viscosity index, flash and fire points, pour point, ash and carbon residue — just to mention a few. By such characteristics, the uniformity of the product and its conformity with an initial batch which had been proven satisfactory in use are established. Thus the principal objective of analyses and tests in manufacturing control is to establish conformity and uniformity of the product.

Product Application

The problems associated with the examination of used lubricating oils are considerably more involved than for new products. Since practically



everything changes on exposure to the oxygen of the air, the oxidized state is thus the natural or equilibrium state. Some things like platinum are oxidized very slowly and protectively; others like china wood oil or linseed oil are oxidized quickly; still others like pyrophoric iron oxidize very rapidly indeed, practically instantly.

Increases in temperature increase the rate of oxidation. Metallic surfaces and salts frequently act as catalysts to accelerate the effects of temperature on oxidation. Engine conditions — with air, high temperature and metallic surfaces — are nearly ideal for rapid and extensive oxidation. It is, therefore, rather remarkable that the oxidation propensities of lubricating oils are effectively retarded by the use of additives.

Figure 10 illustrates the type of changes which generally occur when a straight mineral oil is oxidized. These are noted in oils from actual engine service or in oils from laboratory tests designed to simulate service conditions.

The horizontal axis represents time in service.⁵ Several of the tests which give a direct correlation with the degree of oxidation are shown in the vertical axis. When oils become oxidized, both dissolved (oil soluble) and undissolved matter is formed, as shown on the curves. The former is determined on the clarified oil as the propane insoluble. The undissolved oxidized material is determined as the pentane insoluble of the original sample less the correction for the pentane insoluble of the clarified oil.

For oils which are inhibited against oxidation the trend is similar except that the part of the curve indicating relatively low amounts of oxidized products is extended considerably into the medium or even long "time in service".

⁵"Time in Service" may vary from a matter of hours to several years. It depends upon the quality of the lubricating oil and service conditions such as operating temperatures, contact of the oil with air, composition and operation of metallic engine parts, presence of water or other contaminants, etc.

If the lubricating oil is used in an internal combustion engine it will pick up contaminants which are reaction or decomposition products of the fuel, — gasoline, Diesel fuel or gas, — as well as of the lubricant. Thus, it is normal to find, besides carbon, such contaminants as water and sulfur compounds which result from the oxidation of hydrogen and sulfur. Organic compounds such as alcohols, aldehydes, acids, esters, resins, peroxides, etc. frequently are present in used lubricants, being the result of incomplete oxidation of fuel and lubricant. Nitrogen compounds are also likely contaminants, their origin being nitrogen of the air or nitrogen constituents of some inhibitors and dyes.

In a general way it may be stated that the examination of a used lubricant has several objectives.

First: to establish the identity of the product; specifically is it the product which it is alleged to be?

Second: to establish the degree and nature of the contamination; specifically are there manifest evidences of faulty mechanisms and operating conditions of an abusive nature. Excessive carbon, or gasoline in the lubricating crankcase oil suggests bad rings; radiator antifreeze liquids and water corrosion inhibitors suggest the possibilities of a cracked block or poor gasketing. A high degree of oxidation in the oil suggests excessively high operating temperatures, while excessive dilution indicates unduly low operating temperatures. Totally foreign contaminants, such as linseed oil, commercial abrasives, paint constituents or grease in the crankcase suggest extreme carelessness or even the possibility of malicious contamination.

Third: to establish if the essential characteristics necessary for satisfactory continued use are still possessed by the lubricant; specifically, does it possess the desirable attributes which were deliberately tailored into the original oil, such as viscosity, viscosity index, detergency, non-corrosiveness, etc. and any other special property which for a particular service is uniquely desirable. Reclaiming operations frequently eliminate additives.

Tests on a single used oil sample — whether the tests are chemical or physical — show the "as is" condition of the sample. Even though these indicate the oil to be greatly altered they can seldom predict how this oil will behave in, or affect, a particular mechanical unit. One can only say that an oil in this condition — if apparently unsatisfactory — will very likely adversely affect a machine or engine. More important than a single used oil examination, is periodic sampling and analysis to determine the trend of deterioration or contamination. Badly accelerated degradation is a warning to follow the system more frequently or perhaps even to clean it and change oil. Data from an isolated or spot

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sample is often of little assistance and sometimes even misleading.

Fourth: To resolve complaints and to establish reasons for unsatisfactory performance. This purpose is related to the other three reasons for used oil analysis. It involves not only correct interpretation of test data but also knowledge of engine performance and a review of operating conditions and maintenance habits of the operating personnel.

Clarified Oil

In the examination of used lubricants, it is sometimes desirable to have a portion clarified or freed of undissolved material. Clarified oil is necessary in connection with some analyses which measure characteristics that are likely to be affected by the presence of undissolved contaminants. For example, the presence of water emulsions and undissolved solids affect the values for viscosity, and suspended lead salts cause fictitiously high neutralization numbers. If these interferences are ignored, entirely wrong conclusions will be drawn with respect to the condition of the lubricant.

Before the advent of detergent and dispersing oils, clarification was a relatively simple matter of settling, centrifuging, or filtration through inert materials such as filter paper, absorbent cotton, or finely divided silica. As manufacturers succeeded in

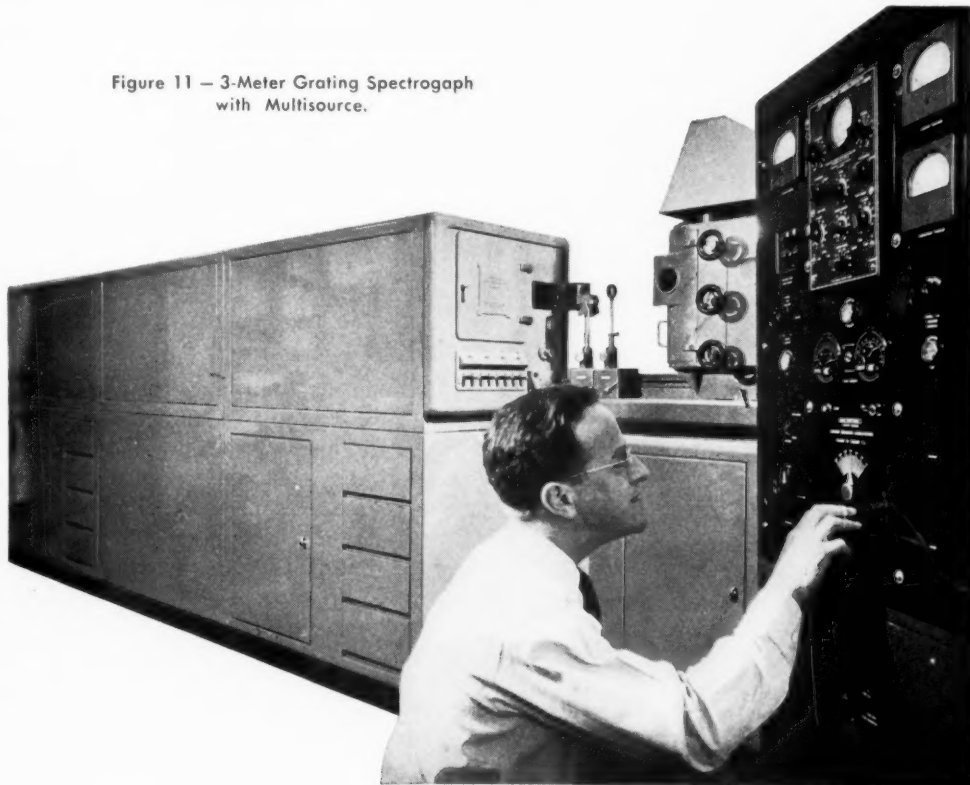
making their oils more effectively detergent, the clarification process became increasingly difficult, so that now it frequently is necessary to resort to more involved techniques which combine the use of wetting agents and solvents with the previous methods.

Dilution

An internal source of contamination in internal combustion engines which tends to reduce viscosity is fuel dilution. A single observation of viscosity could indicate that no change had occurred, when actually thickening due to excessive oxidation had been compensated for by fuel dilution. A distillation test to determine the amount of diluent would eliminate the likelihood of misinterpretation by viscosity alone.

In a modern inhibited Diesel oil, the degree of oxidation is generally low. Frequently fairly good estimation of dilution can be obtained by comparing the viscosities of the new Diesel oil, fuel and used oil. Indications of dilution based on viscosity also should be confirmed with a flash point determination since it is always possible that the reduced viscosity is due to the use of a lower viscosity lubricating oil initially. This is another of the many cases where it is risky to place complete reliance on the outcome of a single test or measurement.

Figure 11 — 3-Meter Grating Spectrophotometer with Multisource.



Contamination

Used motor oil may contain lead, lead oxides, lead halides, lead sulfates and complex combinations of these lead salts. The obvious source of these materials is the tetraethyllead and alkyl chlorides and bromides which comprise tetraethyllead fluid. Additional contaminants are metals, such as iron, aluminum, copper, lead, tin, silver, indium, chromium and their oxides—which originate from engine parts particularly bearings. Some of these constituents may be dissolved in the oil and others undissolved, so here again, clarified and unclarified samples of the used lubricating oils are necessary for a better understanding of the reasons for the condition of the oil. Undissolved metals may indicate defectiveness and mechanical failure of engine parts.

During the winter and spring seasons a contaminant frequently found in used automotive lubricating oil is radiator antifreeze material. Appreciable concentrations of methyl, ethyl and isopropyl alcohols, ethylene glycol and occasionally calcium chloride and glycerine have been found. Their presence suggests a cracked block or faulty cooling system.

Gasket materials, preservative substances including plastic and paint coatings are additional contaminants sometimes present in used lubricating oils. It is important to recognize their presence since often they are clues to sources of trouble. Other contaminants found in used lubricating oils are materials from roads and from dusty atmospheres such as sand, coal, limestone, and sawdust. Some of these materials are particularly dangerous since they are abrasive. In circulating oil systems equipped with water cooling, fresh water is a common contaminant. In marine installations, salt water contamination may occur.

It has been mentioned that carbon is one of the contaminants of used lubricating oils in engine use. Part of this may come from destruction of the lubricating oil but the bulk of it generally arises from blow-by from the combustion chamber. When an effective detergent lubricating oil is used, microscopic examination of the used oil usually shows a uniform distribution of carbon particles over the entire field of vision. A non-detergent oil, in contrast, generally shows a non-uniform distribution of particles and frequently large areas are entirely free of such particles. Microscopic inspection, though not quantitative, often helps to establish the presence of effective detergent quality in used lubricants.

Deposits

Samples of deposits from systems in which lubricating oil has been used are analysed to establish composition in terms of water, diluent, oil, oxidation products, carbonaceous matter, mineral matter,

etc. Constituents are separated and their characteristics determined to establish identity and probable source. Since the amount of oil separated in the course of the analysis of a deposit is generally small, microchemical and other special methods of analysis must be used for its characterization.

Other Methods of Test

The examination of used lubricating oils and deposits requires many other specialized analytical methods and instruments. The emission spectrograph illustrated in Figure 11 is such an instrument. It is used for the qualitative and quantitative determination of metals in petroleum products. Other techniques employed are as follows:

Macro chemical analysis, micro chemical analysis, spot tests, chemical microscopy, electrographic transfer, chromatography, electro analysis, refractometry, colorimetry, flame photometry, polarography, ultraviolet absorption, infrared absorption, X-ray diffraction, mass spectrometry, electron diffraction.

Sampling

With so much time and labor applied in analysis and testing, the importance of proper sampling cannot be over-emphasized. Where the product is a homogeneous liquid the problem of sampling is not too difficult, but where the sample is nonhomogeneous as is the case with used oils and deposits, the sampling should be done as carefully as possible, so that the sample will be truly representative of the material of which analyses is desired.

CONCLUSIONS

The following generalizations are intended to assist in selecting the most appropriate tests for lubricating oils and in deriving the greatest significance from the test results.

Carefully examine the test procedure to determine the lubricating oil properties which are likely to affect the test result.

Select test methods which have a bearing on the objective of the examination. For this purpose consider the service in which the lubricant will be used, the general nature and composition of the lubricant, and the frequency of sampling so that trends in an important property can be recognized.

Remember that progress in the development of new and improved lubricating oils frequently alters the significance of the results by older methods of analysis. Entirely new methods may need to be developed.

PERFORMANCE IN SERVICE IS THE ULTIMATE CRITERION OF SUITABILITY OF A LUBRICATING OIL. THE BEST ANALYSES AND TESTS ARE ONLY GUIDES.

SLUDGE?

NO

RUST?

NO

FOAM?

NO

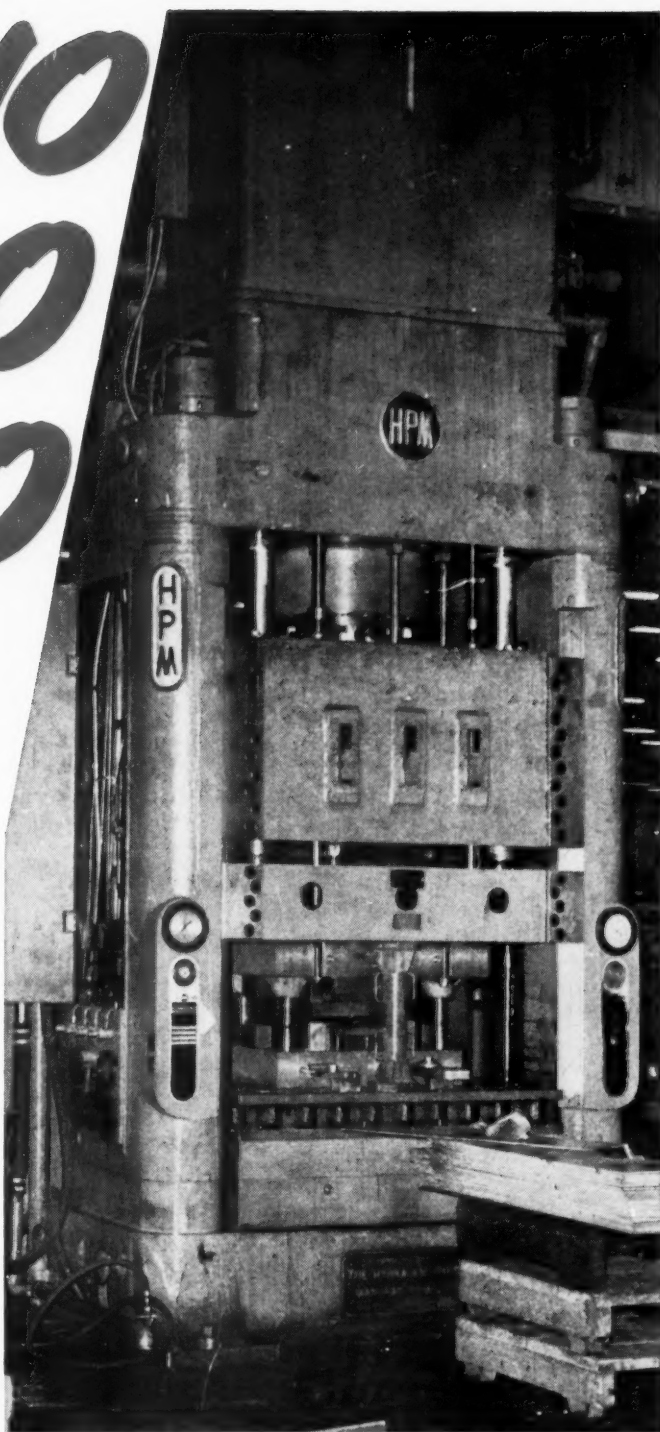
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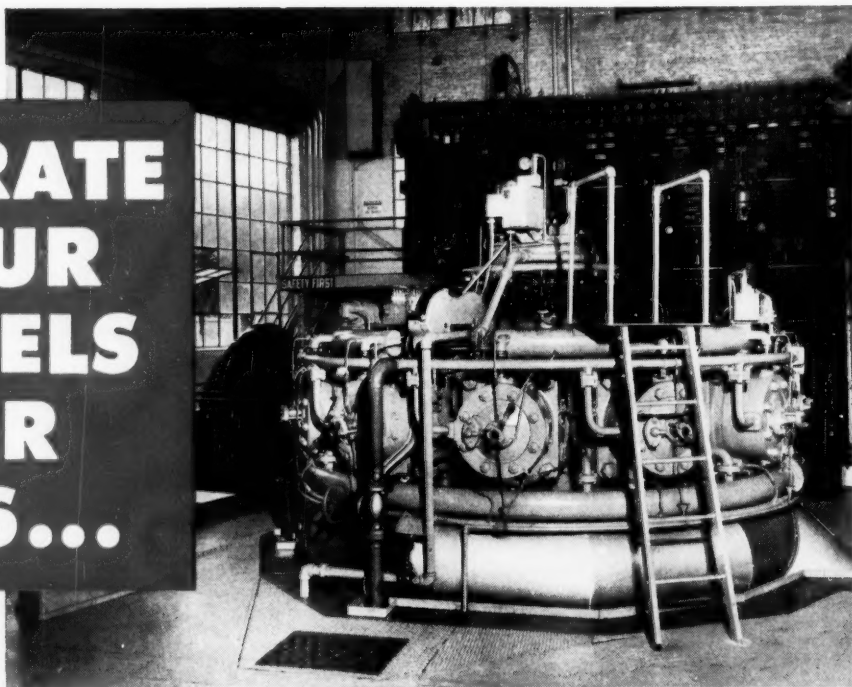
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